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(54) Process for granulating a powder

(57) A process for granulating a powder comprises:—

a) a binding agent (such as starch, carboxymethyl cellulose or guar gum), usually in concentrated aqueous solution, is mixed with a liquid medium, usually an organic liquid, in which the binding agent is insoluble, to form an emulsion;

b) the emulsion is sprayed onto a powder (such as lactose, sucrose, potato starch or a pharmaceutical product) to be granulated; and

c) the liquid medium is evaporated, whereupon the binding agent dispersed in the powder forms agglormeration nuclei around which the powder forms granules.

The process may be carried out in an evacuated vessel into which the emulsion is sprayed by being sucked thereinto; a vessel is illustrated which is provided with mixing and stirring devices and which is swingable so that the drum axis is tiltable anticlockwise and clockwise.

By this process granules can be produced in a closed system without first forming a caked paste.

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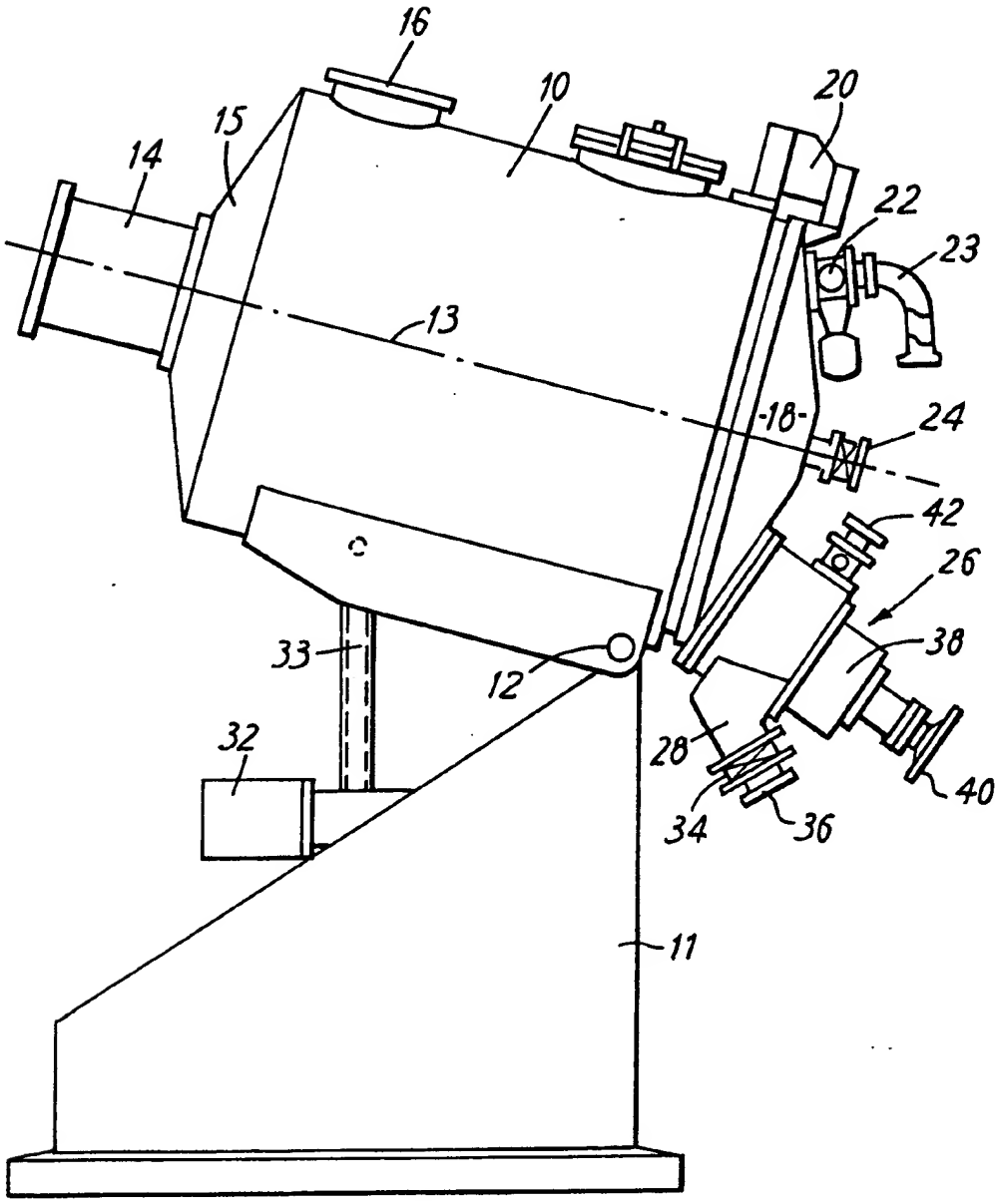
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SPECIFICATION

Process for granulating a p wder

The invention relates to a process for granulating a powder.

With conventional process for granulating a powder, an agglomeration is produced in the powder. Starch paste in particular is still a frequently used medium in this process, which however has the disadvantage that the prepared starch paste produces a high viscosity solution which requires much stirring in order to be dispersed in the mixture. At the end of this known process a cake-like agglomeration mixture is formed which must be dried in large lumps (in a granulate flash-drier) and then dispersed.

It is an object of this invention to provide a process by which qualitatively substantially improved granules can be produced in a closed system without creating a caked agglomeration paste as intermediate product.

The process of the invention comprises the following steps:

a) a binding agent is mixed with a liquid medium in which the binding agent is inssoluble, to form an emulsion in which the binding agent is dispersed;

b) the emulsion is sprayed onto the powder to be granulated, whereby the binding agent is dispersed in the powder; and

c) the liquid is evaporated, whereupon the binding agent dispersed in the powder forms agglomeration nuclei around which the powder agglomerates to form larger structures, i.e. granules.

The essence of the process consists in mixing a concentrated aqueous solution with an organic liquid which will not dissolve in the solution, in order to ensure an emulsion-like consistency of the mixture. For if the binding agent were to dissolve in the organic liquid medium, the agglomeration nuclei could not be localized and fixed and after dispersing the solution in the power undesirable "caking" would occur.

Powders which can be granulated include lactose, sucrose, potato starch and pharmaceutical products such as phenacetine, Suitable binders include starch, carboxymethyl cellulose and guar gum, each in aqueous solution of paste. Suitable liquid media are organic liquids such as isopropanol, methylene chloride, chloroform, carbon tetrachloride and trichlorethylene. The concentration of aqueous binder solution or paste must be selected to obtain the optimum suspension. As larg as possible amount of binder should be used.

It is particularly advantageous if th powder is kept in suspension in an evacuated vessel and the mulsion is sprayed on by being sucked into the vessel. A suitable such vess l is one in which is arranged a driven shaft extending in the drum axis and carrying mixing, stirring and conveying devices, and which is swingable in the vertical plane c ntaining the drum axis in such a way that the drum axis is tiltable anticlockwise and clockwise towards its horizontal position, and the mixing and conveying devices are arranged and driven in such a way that for each tilt of the drum they convey the powder upwards against gravity, away from the respective drum wall lying underneath.

The invention is described with reference to the accompanying drawing wherein the single Fig. is a side view of such suitable apparatus for carrying out the process. This equipment is claimed in my copending application filed today corresponding to Austrian Application No. A-6110/80.

A cylindrical drum 10 is rotatably hinged about a horizontal swivel axis 12 and whereby it is supported on a base 11. A rotary shaft (not shown) extending along the axis 13 of the drum is driven from a motor 14 attached at one end 15. The drum may have a double wall to allow the contents to be heated, and attached to the rotating shaft are mixing, stirring and conveying spirals or screws such as are described in German OLS No. 2,218,729 or the corresponding U.S. Specification No. 3,946,996 of which I am the inventor. The drum 10 is swingable about the axis 12 by means of a motor 32 and linkage 33. A lid 18 hinged to the drum at 20 facilitates access to the drum for cleaning and repairing purposes. The lid 18 is provided with three openings for loading and unloading the drum contents. The inlet valves 22 and 24 are respectively for admission of powder (via hose 23) and liquid matter, in each case this is preferably effected by sucking the material into the evacuated drum. The valve 24 leads to a nozzle (not shown) within the drum. The sieving unit 26 equipped with a motor 38, and outlet pipe 28 and an outlet valve 34 and flange 36 and additional inlet 42 is provided for the purpose of sieving and removal of the product and is described in detail in the aforesaid two specifications and also in my copending application filed today corresponding to Austrian Application No. A-6112/80 which describes control wheel 40 for a disc valve closing off the unit 26.

At the upper end of the drum, remote from the lid 18, the equipment is preferably provided with a flange or similar arrangement forming the connection 16 for a conventional vacuum-producing and controlling system.

The procedure for carrying out the process according to the invention with starch as binder and using this equipment is as follows.

A starch solution is initially prepared in the usual way be mixing it cold and then heating it. It is then emulsified in a liquid, e.g. a mixture of isopropanol and methylen chloride. In the emulsion produced concentrated

starch paste particles are dispersed in th form of dr plets in the liquid, just as the solv nts disperse in the form of dropl ts in the solution phase. (Similar to oil/water and water/oil mixtures in creams.)

After powder to be treated has been loaded into the drum 10, the emulsion obtained in this way is sprayed onto the powder mixture, whilst the powder is preferably held suspended in the drum by the mixing and conveying devices. During this process the emulsion of starch paste is dispersed as droplets in the powder mixture, whereupon the liquid medium (methylene chloride and isopropanol) is evaporated and the starch paste droplets are localized. These starch droplets now represent agglomeration nuclei comparable to crystallization nuclei, around which the other powder particles agglomerate to form larger structures. Due to the fact that the mixing and conveying devices are arranged and operated under the invention in such a way that for each tilt of the to-and-forth swinging drum 10 they convey the powder upwards against its gravity, away from the respective drum wall lying underneath, compacting is avoided during mixing so that the floating or almost constantly free-falling agglomeration nuclei may be freed of their residual moisture by e.g. vacuum flash drying. An aqueous solution of sorbitol may be used successfully in place of starch paste.

The invention will now be further explained by way of two examples, which are carried out in the apparatus shown in the drawing.

Example 1

50 g of corn starch are mixed with 25 ml of water and then combined with 50 ml of isopropanol and 20 ml of methylene chloride in a high-speed agitator. A low-viscosity emulsion is obtained. If the powder mixture contains 80% lactose and 20% starch, it is advantageous to emulsify only 5% of the starch in the manner described and to add the remaining 15% of the starch dry together with the powder. Whilst the drum together with the powder mixture it contains is swinging in the manner described about its horizontal position and whilst the mixing and conveying devices ensure that the powder is kept in constant supension or free-falling, the starch emulsion is sprayed onto the powder.

Compared with conventional processes this process consumes much less starch paste and ensures a substantially improved dispersion of the binding agent.

In a 600 1 vacuum apparatus as shown in the drawing are introduced 250 kg lactose, heated to 60°C and in vacuum moistened with a 10 kg maiz starch previously b il d with 5 1 water and aft r c oling dispersed in 10 1 isopropanol and 2 1 methylene chloride. The solvents are, under mixing and swinging, evaporated under vacuum and the r sulting granules are discharged through the attached sieving unit 26 of the apparatus.

Example 2

A mixture of 90% lactose and 10% sorbitol is granulated. For this a 10% aqueous sorbitol solution is prepared (9 parts sorbitol, 1 part water), and, once dissolved, emulsified with 1 part isopropanol and 0.5 parts methylene chloride in a high-speed agitator. By using the process described above the same effect is obtained: the low-viscosity solution of sorbitol is at first not able to stick since it is prevented from doing so by the isopropanol and the methylene chloride. Therefore the solution is dispersed in the lactose powder mixture without agglomeration taking place immediately. Only when the methylene chloride and the isopropanol have been dried off (e.g. by vacuum flash drying) becomes the remaining water with the sorbitol very sticky. But since it has already been finely dispersed as droplets within the powder, a granulate with superior qualities is automatically produced.

CLAIMS

1. A process for granulating a powder which comprises the following steps:—

a) a binding agent is mixed with a liquid medium in which the binding agent is insoluble, to form an emulsion in which the binding agent is dispersed;

b) the emulsion is sprayed onto the powder to be granulated, whereby the binding agent is dispersed in the powder; and

c) the liquid is evaporated, whereupon the binding agent dispersed in the powder forms agglomeration nuclei around which the powder agglomerates to form larger structures, i.e. granules.

2. A process as claimed in Claim 1, wherein the binding agent is starch, carboxymethyl cellulose or guar gum.

3. A process as claimed in Claim 1 or 2, wherein the liquid medium is isopropanol, methylene chloride, chloroform, carbon tetrachloride or trichlorethylene.

4. A process as claimed in Claim 1, 2 or 3, wherein the powder to be granulated is kept suspended in an evacuated vessel and the emulsion of binding agent is sprayed on by being sucked into the vessel.

5. A process as claimed in any preceding claim, wherein the dispersion of the binding agent in the powder takes place in a vessel equipped with mixing and conveying means and which is tiltable so that the powder is constantly urged upwardly.

6. A process as claimed in Claim 5, which is carried out in apparatus substantially as herein described with reference to r as shown in the accompanying drawing.

7. A process for granulating a powder, substantially as hereinbefore described with reference to ither of the Examples.

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